The Chemical Age

A Weekly Journal Devoted to Industrial and Engineering Chemistry

BOUVERIE HOUSE, 154, FLEET STREET, LONDON, E.C.4

Telegrams: ALLANGAS FLEET LONDON — Telephone: CENTRAL 3212 (10 lines)
GLASGOW: 116, Hope Street (Central 3970) — BIRMINGHAM: Daimler House, Paradise Street (Midland 0784-5)
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Cement from Slag

THERE appears to have been a good deal of muddle in regard to the supply of cement which led to a serious shortage last summer. We are now informed officially that there is as much cement as can be required by anyone, but whether the next building season will see a new cement shortage appears to be a subject upon which there is considerable divergence of opinion. The official view is that labour during that season will be so short that there will be plenty of cement for a restricted programme. The industry, on the other hand, appears to consider that this shortage of labour will not materialise, but that the cement requirements will depend on the Government's A.R.P. shelter policy and on the extent to which a rehousing programme is put into operation. The industry is keenly interested in increasing the quantity available and appears to be looking to the blast furnaces to provide plenty of raw material in the form of slag.

Blast-furnace slag has not been as much used in this country as it has been abroad, because we have here an abundance of clays from which Portland cement can be made so cheaply that slag manufacture has been rather discouraged. The Ministry of Works maintains that there is very little suitable slag available, that satisfactory slags can only be produced from furnaces working on hæmatite ore, and that in any event its use for producing blast-furnace cement would not increase the total cement supply by more than 6 per cent. This view is controverted by cement experts, who believe that the available total of suitable blast-furnace slags is at least ten million tons a year, the use of which, it is contended, would revolutionise the industry. The Ministry believes that the grinding of slags would in any event be a bottle-neck preventing expansion, whereas the experts point out that there is plenty of grinding plant available, much of which lies idle in winter and could then be employed in grinding slags for later use. Moreover, very little steel would be required to re-equip a round score of abandoned works with ball mills. The view that only hæmatite slags are suitable also does not commend itself to the cement industry.

A path through this maze of conflicting views may be gained by considering the manufacture of cement from blast-furnace slag. The quantity of slag available when making iron from hæmatite ores is from 1200 to 1400 lb. per ton of iron; from foundry iron it is 2400 to 3000 lb. per ton; from basic iron 2500 to 3200 lb.; and from Cleveland ores 3200 to 3600 lb. A good deal of the slag is made into road material, railway ballast, or slag bricks, which depend for their effectiveness upon hardness of the slag. When the slag is poured into a 5-ton ladle it takes about 12 to 14 hours to cool and then slakes freely and is not suitable for building purposes. More rapidly cooled, however, it furnishes a hard resistant material. On slow cooling the calcium bisilicate (CaO, SiO_2) first formed is converted into the orthosilicate ($2CaO$, SiO_2). This, on slow cooling, passes through the α-form into the β and thence into the γ-form, a passage that is accompanied by a large increase of volume setting up severe stresses within the material, and resulting in disintegration.

A century of attempts to make blast-furnace cement has shown that the only successful process for manufacturing cement equal in quality to Portland cement is by making a true cement clinker from limestone and granulated slag. If the slag is required for this purpose the burden charged into the blast furnace can be so modified as to make a slag of suitable composition, the only essential being that the slag shall carry away all the impurities from the iron. Frequently the slag is run into a rapid stream of cold water by which it is immediately obtained in the granulated form. Usually, however, in this country it is discharged into ladles and is thus subjected to slower cooling and must be crushed before use. Portland cement consists primarily of alite, a solid solution of $3CaO$, Al_2O_3 in $3CaO$, SiO_2, and celite, a solid solution of $2CaO$, Al_2O_3 in $2CaO$, SiO_2. The setting and hardening depend on the decomposition of alite by water, to form hydrated calcium silicate and aluminate. The problem is then to bring blast-furnace slag to the composition of Portland cement. The process is essentially as follows: limestone and slag are separately crushed and dried, then mixed and ground together in the correct proportions. This raw meal is burnt to clinker in a kiln. The clinker is then mixed with a suitable proportion of granulated slag and ground to cement fineness, the slag being added to provide sufficient active silica, since there is otherwise on hydration an excess of lime. It is evident that, provided the slag is reasonably true to its usual composition, and is produced either granularly or in the softer form suitable for grinding, it can readily be used for the manufacture of cement, and can be made to conform to the British Standard Specification in that the ratio $CaO/(SiO_2 + Al_2O_3)$ is between 2 and 2.5. The early strength of blast-furnace cement may often be low, but it readily hardens and will prove just as useful a material for building purposes as the true Portland cement. It would be a help to the iron industry if this outlet for the slag could be adopted as regular practice.

The Chemical Age Year Book

THE first entry in the Fleet Street Book of Rules reads: "Never complain nor explain." This is an admirable injunction for normal working in quiet times. The last few months, however, have been neither normal nor quiet, and it must have been the experience of every one of us that every rule has its exceptions in war time. THE CHEMICAL AGE even now has no complaint to make, but it has an explanation to offer. It has succeeded amid unimaginable difficulties in maintaining its times of publication with unfailing success except in one particular. The publication of *The Chemical Age Year Book* is some days behind schedule, and this is a matter on which we think it right to take our subscribers into our confidence. We do so the more readily as the story is one which redounds to the credit of all concerned, more particularly the printers. It is in fact one of the many fine pages in the still unfinished chapter of the unscrupulous attempt of the enemy to dislocate, if not to destroy, British trade and industry. The background is the *blitzkrieg*, and the outlines of the story can be indicated best, without giving anything away to the enemy, by an account of the chief difficulties which the publishers of THE CHEMICAL AGE have had to face as a group.

Benn Brothers, Limited, publish not only *The Chemical Age Year Book* but three others of a similar character. The final stages are placed in the hands of various printing firms, and it is their *blitzkrieg* troubles which have caused nine-tenths of the delay. The premises of one of these firms have been almost completely destroyed. There were in those premises many hundreds of blocks required for one of the year books. The firm had suffered such serious damage in earlier raids as to cause a transfer by mutual arrangement of the year book to another printer, and the blocks were providentially saved with only a few days to spare. But the combination of these various circumstances meant an inevitable slowing down of the work.

Another printing firm in an entirely different area was also reduced to ruins. In this case, unfortunately, year-book work was in active progress, and material accumulated for the publication was destroyed. For example, the entire stock of special paper, collected and prepared for the diary pages which have formerly been a part of the year book, went up in smoke. Much of the editorial "copy" was already in the hands of these printers, and its destruction meant that the work had to be done all over again, including, in some cases, the reference of important matter to firms and institutions associated with the industry. Other firms lost time through temporary evacuation of their premises on account of unexploded time bombs in their immediate neighbourhood. The temporary failure of gas and electricity supplies added to the difficulties. Another, though an indirect, hindrance was the requisitioning of the premises of two printing firms by Government order, with the consequent transfer of their activities at short notice to other points.

This recital is not intended to imply that printers have suffered more severely than other sections of the industrial community. What emerges is that they have proved themselves capable of withstanding, on their particular ground, the full blast of the *blitzkrieg*. They have faced, with great courage and adaptability, obstacles never before encountered, and they have triumphantly surmounted them. The only result of the German attack on this vital service has been a slight retardation instead of the absolute stoppage which they sought to enforce. *The Chemical Age Year Book* is now complete and is emerging from the binders' hands. It will be in the hands of the industry in a very few days. Its appearance is the best guarantee the publishers can give of their inflexible determination to carry on their activities on behalf of the chemical industry no matter what pressure the enemy may bring to bear.

The publishers offer this explanation with the more confidence as it is obvious that many of our readers have had to face even more serious troubles than they. The chemical industry, in common with other national services which have been the target of German air attack, will assuredly be the first to take pride in a record of achievement which has been matched time and again in its own experience.

Notes and Comments

Manufacturers Patent Rights

THE war has brought in its train a multitude of problems for industry generally and some of these, where their effect is widespread, have been solved by the passing of appropriate legislation. At the same time there are other war-time problems that are less general in their implications, but are none the less worrying to those sections of industry to whom they are peculiar. One such is the loss to chemical engineers of the cover period for their patent and other registration rights, due to their plants having been put to work of national importance for the duration of the war. Among such manufacturers there is a growing feeling, which we readily support because of its reasonableness, that the Government should arrange for an automatic extension of the cover to include the period thus put in abeyance. A condition, which would cheerfully be accepted by the manufacturers, could be the payment of a fee by applicants. So far as patent and other registration rights in this country are concerned an arrangement such as that suggested could be put into effect with a minimum of delay. There are, however, many patents which have been taken out in the Dominions and Colonies and extensions in these cases would, of course, take far longer to arrange. Nevertheless, it should be borne in mind that overseas authorities usually follow the procedure of the Mother Country in deciding such questions, so that a ruling here would be of great importance.

Beryllium

FEW industries can show such a percentage of improvement in 1940 as the beryllium field, it is noted in *Mineral Trade Notes* of the U.S. Bureau of Mines. Insignficant a decade ago, American sales of this remarkable metal and its products have grown so that in 1940 they may amount to $500,000 or more. A fair amount of beryllium oxide is sold—chiefly as a super-refractory but also for other purposes—but it is used mainly in alloys, almost entirely in beryllium copper. Beryllium nickel, however, also has possibilities, aluminium alloys with beryllium additions are being investigated, and the metal itself has recently been made experimentally in the United States, to sell at substantially less than the former price of German made metal of less purity. The recent development of beryllium alginate as a material for textile fibre should afford yet another outlet for this extremely versatile element.

Synthetic Production of Phenol*

Raschig Process Employed in Large American Plant

DESCRIBED as the largest of its kind in the world, a new phenol plant, in which the Raschig process is in operation, has recently been opened by Durez Plastics and Chemicals, Inc., as a unit of their large plastics plant at North Tonawanda, N.Y. It is considered that the new plant will not only assure an independent and controlled source of the purest phenol obtainable in the country and make possible greatly improved plastics, but will also be of importance in view of the use made of phenol in certain explosives.

The advantage claimed for the Raschig process is that it produces the highest grade of phenol with a minimum of by-product. Processes heretofore employed produce from two to five pounds of by-product to one pound of phenol, while the Raschig process develops less than one-tenth of a pound of by-product for each pound of phenol. The Durez new plant has a capacity of 15 million pounds a year.

The process is so intricate that many leading chemical engineers in America refused to consider it when it was first offered there a few years ago. First a vapour mixture of benzene hydrochloric acid and air is passed through a catalyst. This produces a mixture of chlorinated benzenes from which monochlorbenzene is distilled in the pure state for use in the second step; and, secondly, a vapour mixture of monochlorbenzene and steam is passed through a catalyst which produces phenol and regenerates the hydrochloric acid. These two stages form a completely continuous process, and during the process most of the materials formed and the catalysts are recovered for re-use.

Description of the Operation

Invented by Dr. W. Prahl and Dr. W. Mathes of the Dr. F. Raschig G.m.b.H. of Ludwigshafen, Germany, in 1930, the process functions in two catalytic vapour-phase stages. In the first stage, benzene is chlorinated with hydrochloric acid and air, in the presence of a catalyst, with the production of monochlorbenzene. In the second stage, monochlorbenzene is hydrolysed with water, in the presence of a catalyst, with the production of phenol and hydrochloric acid. The first stage reaction proceeds at about 230° C., is exothermic, and is represened by the following equation :

$$C_6H_6 + HCl + \tfrac{1}{2}O_2 = C_6H_5Cl + H_2O$$

As only about ten per cent. of benzene is converted in one pass through the catalyst and as small quantities of polychlorbenzenes are formed, and as, also, a trace of oxidation takes place, the actual products of the first-stage reaction are: C_6H_6, C_6H_5Cl, polychlorbenzenes, CO_2, CO, N_2 and H_2O, so that several orderly steps are required to effect the separation and recovery of the products of the reaction. This is accomplished by continuous fractional condensation, whereby the chlorinated benzenes and part of the unreacted benzene are collected in liquid form for the separate recovery, by continuous fractional distillation, of benzene, monochlorbenzene and a mixture of the polychlorbenzenes. A part of the balance of unreacted benzene vapour is condensed for re-use, in a final condenser, while the remainder is recovered by scrubbing the tail gases with an organic absorbing liquid from which benzene is continuously distilled and condensed for re-use.

In practice the first-stage system operates by drawing the first-stage reactants in the vapour state, at slight negative pressure, in succession, through contact chambers, a fractional condensing tower, and a final condenser. When conversion to form first-stage products takes place, these products condense and are removed from the fractional condenser, which operates at a temperature slightly above the boiling point of benzene. The benzene vapours which pass from the fractional condenser are then condensed and recovered from the final condenser and tail gas recovery system.

The second-stage reaction proceeds at about 425° C., is endothermic, and is represented by the following equation :

$$C_6H_5Cl + H_2O = C_6H_5OH + HCl$$

As only about ten per cent. of the monochlorbenzene is converted in one pass through the catalyst and as more water is present than given by the equation, and as, also, traces of side-reaction products are formed, the actual products of the second stage reaction are C_6H_5OH, HCl, H_2O, oxydiphenyl and diphenyl ether, so that several orderly steps are required to effect the separation and recovery of these products. By heat interchange between the sensible heat of the products of the reaction and the azeotropic vapour mixture of the reactants, the products are first cooled to about 200° C. and then further cooled in a fractional condenser by being required to vaporise a liquid mixture of the reactants. Next by further fractional condensation a water solution of HCl (15 per cent.) containing about 5 per cent. of the phenol formed, is condensed and recovered. Leaving the fractional condenser the vapours contain about 95 per cent. of the phenol formed, together with all the azeotropic mixture of the reactants, from which the phenol is dissolved by scrubbing with hot water under temperature conditions such that the azeotropic vapour mixture of the reactants remains in the vapour state to be later heated to reaction temperature and passed through the second-stage contact chambers.

Use of Heat Interchange

The products of the second-stage reaction are thus collected in the form of a recovered HCl solution (15 per cent.) containing dissolved phenol and a water solution of phenol. These phenol solutions are separately extracted, in continuous counter-current fashion, by benzene, which extraction step constitutes the entry point for the benzene required in the first-stage reaction. After benzene extraction to remove phenol the recovered HCl solution passes to HCl vaporisers to supply that reactant of the first-stage reaction while the extracted water solution of phenol is reheated, stripped of benzene, which is condensed and recovered, and then cycles back to the phenol scrubber to absorb more phenol.

The combined benzene-phenol solutions from the extraction steps pass to a continuous fractionating still. The distillate from this column delivers benzene vapour to constitute that reactant of the first-stage reaction, while the outflow from this column consists of crude phenol (97 per cent.) which is subsequently recovered in pure state by continuous vacuum distillation.

In practice the second-stage system operates by cycling the reactants in vapour state, at slight negative pressure, in succession, through contact chambers, heat interchangers, a vapour superheater, a fractional condensing tower and a scrubbing tower. When conversion to form second-stage products takes place, these condense and are collected from the fractional condenser and the scrubbing tower. Throughout the second-stage system efficient heat-interchange principles are utilised to avoid condensation and re-evaporation of the reactants and to utilise the sensible heat of the products of the reaction. An examination of the two reactions, above, will disclose the production of phenol from benzene and air by utilising hydrogen chloride, in a sense, as an oxygen carrier.

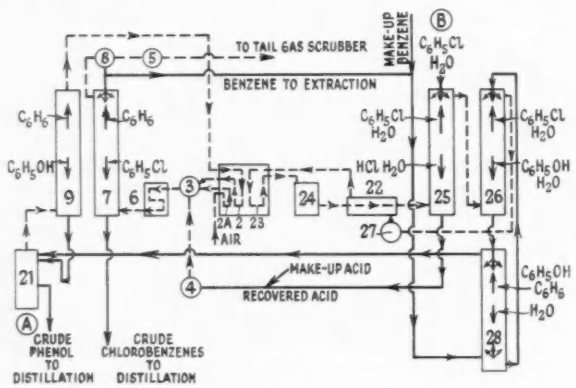
From this brief description of the process, which is continuous and which involves a regenerative cycle with respect to hydrochloric acid, it would appear that the many successive steps necessary require an elaborate system of exact control and synchronisation. Such is not the case in practice; on the contrary, in both stages of the process the reactants in the vapour state are caused to move in succession through contact chambers, heat interchangers, or heat recovery apparatus, a fractional condensing tower, a final condenser, or its counterpart, and vaporisation apparatus. Only when

* From an article in *Chemical Industries*, 1940, *47*, 6, pp. 522-526.

conversion takes place do the products of the reactions condense and flow from the condensing equipment to be collected. If no reaction took place, steam, water and electrical energy would be expended only for vaporising, moving, and condensing the reactants, but the reactants would be substantially recovered in original form, minus, of course, mechanical losses.

Reference to the flow sheet will clarify the operation of the process. Liquid flows are solid, vapour flows are dotted.

Starting at (A)—benzene is being boiled in a steam heated tubular evaporator (21) from a mixture of benzene and phenol and is passed into fractionating column 9. The benzene vapour passes through a tubular heater 2, in the vapour superheater, and then into vapour mixer 3. Hydrochloric acid is boiling from 4 into vapour mixer 3 and the air is being drawn into vapour mixer 3 through tubular heater, 2a, in the vapour superheater. The mixture of these three vapours in proper proportion and at a temperature of about 205° C. passes into contact chamber 6, where chlorination takes place. The products from chamber 6 pass into scrubbing tower 7 wherein condensation to liquid form takes place to form crude chlorobenzene which flows from the bottom of tower 7 to storage for subsequent continuous fractional distillation. Above tower 7 is condenser 8, which condenses benzene vapour driven off in the tower. Part of this distillate is used as reflux for the tower while the balance is used to extract phenol in the second stage. Item 5 is a suction fan which draws the reactants through the first-stage system. This fan discharges into a scrubbing tower in which benzene is absorbed from the tail gases by a hydrocarbon oil from which the dissolved benzene is continuously distilled for reuse.



Flow Sheet of Raschig Synthetic Phenol Process

Starting at B, a mixture of pure chlorobenzene and water is added into the top of scrubbing column 25 within which the products of the sceond stage reaction are circulated by a turbo-blower 27. The temperature conditions in column 25 are so maintained that the azeotropic mixture of chlorobenzene and water is formed to augment the vapours leaving the column. The excess water added at this point absorbs the HCl from the vapours and the recovered HCl solution (15 per cent.) flows from the bottom to be vaporised in the first-stage system at 4. The vapours leaving the top of column 25 consist of chlorobenzene, water and phenol and pass into the bottom of the phenol washer 26, which is refluxed with hot water to dissolve out the phenol. This is operated at such a temperature that the azeotropic mixture of chlorobenzene and water remains in the vapour state. The phenol-water solution leaving the bottom of column 26 is passed through extractor 28, wherein the phenol is extracted by benzene. The phenol-benzene extract from the top of extractor 28 flows to evaporator 21 to supply the benzene vapour for the first stage while the crude phenol from the evaporator is sent to storage for subsequent purification by continuous vacuum distillation.

The chlorobenzene-water vapour leaving the top of column 26 is picked up by turbo-blower 27 and passed through tubular heat exchanger 22 to be raised in temperature by absorbing heat from the hot products of the second stage reaction coming from contact chamber 24. Leaving exchanger 22 the reactants lack about 100° C. in temperature so they are passed through tubular heater 23 in the vapour superheater and then directly into the second stage contact chambers 24. Leaving chambers 24 the hot products exchange sensible heat with the incoming reactants and are thereby partly cooled. From the heat exchangers 22 the products go directly to the bottom of the acid recovery tower 25.

Advantages of the Process

The process is of particular interest with respect to the following:—

1. For all practical purposes the process is regenerative with respect to hydrochloric acid as indicated by the equations and as evidenced by the fact that the normal operating efficiency of the HCl recovery cycle is about 97 per cent. This efficiency includes loss of HCl in the form of higher chlorinated benzenes and all mechanical losses in handling.
2. The process forms by-products to the extent of only one-tenth of a pound per pound of pure phenol produced, these being small quantities of polychlorbenzene, diphenyl compounds and tarry matter.
3. The first stage catalyst is truly selective towards benzene in that unsaturated compounds or toluene are not chlorinated but are oxidised to CO_2 and CO; thus no trace of cresols exists in the phenol, which has a solidification point of 40.70° C. minimum.
4. Equipment items handling hydrochloric acid liquid or vapour are so designed that maintenance is reduced to a figure not above the maintenance encountered in the average chemical plant.

Hydrogenation of Glucose to Sorbitol

Pressure Method Examined

CONVERSION of glucose into sorbitol may be effected either by electrolytic reduction (a process in large-scale operation in the United States) or by pressure-hydrogenation in presence of a nickel catalyst, though the latter method does not appear to have attained the importance of the electrolytic method. In an investigation of the conditions for hydrogenation of glucose under pressure, S. Borisoglebskii (*J. App. Chem.*, U.S.S.R., 1940, 4, 571) started from a 70 per cent. solution of edible glucose which was hydrogenated in a horizontal autoclave fitted with stirring gear in presence of a nickel catalyst (prepared by treating a nickel-aluminium alloy with caustic soda). He claimed to obtain a 99 to 100 per cent. conversion to very pure *d*-sorbitol by working at a hydrogen pressure of 60 to 80 atm. and a temperature of 100 to 120° C., while maintaining the reaction solution at a *p*H of 8 to 8.4 and using 2 to 3 per cent. of active catalyst.

Chemical Engineer's Claim Fails

Scrap-Metal Firm's Generosity

JUDGMENT was found for the defendants, Messrs. E. Hind and Co. (South Bank), Ltd., scrap merchants, on Thursday last week by Judge Gamon, in a case brought against them by Dr. Otto Einerl, a Vienna-born chemical and metallurgical engineer, who came to Britain in 1939 and was recently released from a British internment camp. Dr. Einerl sued for two weeks' alleged arrears of salary, and other expenses, his claim amounting to £200, but the Judge ruled that he had not established his claim. The plaintiff broke down in tears when judgment was pronounced, and Judge Gamon suggested that the company should not ask for costs. Mr. Fenwick, for Messrs. Hind, said that not only were they not asking for costs, but that if Dr. Einerl would call on the company's solicitors he would receive a substantial *ex gratia* payment.

Fire Fighting

Details of the Government Scheme

THE Home Secretary and Minister of Home Security (Mr. Herbert Morrison), in announcing the Government's new compulsory scheme for fire-bomb fighting, explained that by three new Defence Regulations he was taking power to impose compulsory part-time service in all branches of the Civil Defence Force for men and women between the ages of 16 and 60. For the time being, this power would be used only in connection with fire-prevention service for countering fire-bombs, and the Orders made under the new regulations would be restricted to this form of service and, again for the time being, would apply to men only, within age limits which the Minister would determine.

The amount of compulsory service required is not to exceed 48 hours per month. The compulsory order will apply in all those areas where the risk of fire raids is sufficiently great to require it. It is proposed to ask the Regional Commissioners to select those local authority areas in their regions which are the proper ones for the application of the new order.

Industrial and Commercial Premises

Under the scheme for industrial and commercial premises, all occupiers of factory and office buildings in areas to which the order applies are required to make satisfactory arrangements for fire prevention, and in particular to ensure that enough people are available, each with his allotted task. The number required for premises of given size or character will be indicated in a series of practical rules for fire-fighting in factory and office premises, which will be issued by the various authorities responsible. There is a general obligation upon men of all grades, including management, in commercial and industrial premises, to take their turn of duty in fire-bomb fighting outside their normal working hours. For this service they will not be entitled to remuneration. But if proper and adequate arrangements to the satisfaction of the responsible authority can be made on a voluntary basis (paid or unpaid) the general obligation need not be enforced. If arrangements, either on a voluntary or compulsory basis, are not made by the occupier, the appropriate authority is empowered to step in and make them, charging the occupier with any expenses involved. Compulsory service at the place of work takes precedence over compulsory service at the place of residence, but those already doing 48 hours' civil defence service a month in their residential area are not liable to be called upon for duty at their place of work.

The directions to an occupier will come from the appropriate authority which will vary according to the class of premises. The Ministry of Labour will be responsible for the general body of factories employing more than 30, and the local authority for factories employing less than 30 and for commercial premises. In the case of small factories, shops, etc., the local authority, which will be the responsible authority, can require premises to combine if their numbers are too small for them to have an effective independent fire-prevention organisation. If necessary, a separate or combined organisation may be reinforced by the local authority from its general pool of enrolled personnel.

The age limits for fire-prevention duty in industrial and commercial premises will be laid down by the Minister of Home Security, within the general limits allowed by the Regulations. There will be a right of appeal on grounds of medical unfitness or excessive hardship, to tribunals of three.

Described as an economy issue, No. 27, Vol. IV, of the Wild-Barfield Heat-Treatment Journal, published by WILD-BARFIELD ELECTRIC FURNACES, LTD., Watford, contains articles on "Delaying Age-hardening of Duralumin," "The Care of Pyrometers" and "Tongs and Tools for High-speed Steel Hardening."

New U.S. Commercial Minerals

Pinite and Hectorite

PINITE, a microcrystalline form of mica of the muscovite family, closely related to sericite except for its smaller crystal size, has appeared on the scene as an industrial mineral. The divisional chief engineer of the U.S. Bureau of Mines visited the mine operated by the Stockton Firebrick Co. near Rochester and Oreana, Nevada, where a considerable deposit of favourable characteristics has been opened. The plant of the same company, at Pittburg, California, was also visited, and commercial shapes intended for kiln linings and linings of coolers were seen in course of preparation. These exceedingly hard, abrasion-resistant bricks and blocks have won a place for themselves as a special super-refractory.

Another exceedingly interesting mineral is the Hector clay or hectorite mined by several companies near Hector, California, east of Barstow, on the Atchison, Topeka, & Santa Fé Railway. The pure clay mineral is a hydrated magnesium silicate, which is waxy when wet but crumbles to a dazzling white powder. The Hector clay is usually mixed with finely divided calcium carbonate, most of which will settle out from a dilute suspension in water. The clay is mined under lava-flows from the Pisgah volcano, and the principal producer, California Talc Corporation, a subsidiary of the National Lead Co., is marketing it as a high-grade oil-well drilling mud, in which capacity it fulfils the functions of a bentonite (U.S. Bureau of Mines, R.I. 3538).

Maleic Acid Manufacture

Furanes as Raw Material

WHILE to-day it is usual to employ aromatic ring compounds like benzene and naphthalene in the manufacture of maleic acid by oxidation processes, it is well known that many aliphatic and heterocyclic bodies likewise yield maleic acid among their products of oxidation. Furfural and other compounds containing the furane ring have thus engaged the attention of research workers as likely raw materials for maleic acid, a material which is rapidly growing in importance as an intermediate for synthetic resins. It is now claimed that exceptionally good yields of maleic acid can be obtained from dihydro- and tetrahydrofurane by oxidation in the vapour phase at 250° to 450° C. in presence of a vanadium or molybdenum oxide. Details are published in U.S.P. 2,215,095 of the General Aniline and Film Corporation (Convention Date, Germany, December 12, 1938). Yields of maleic acid (as the hydrate) from tetrahydro- and dihydrofurane are stated to amount to 80-90 per cent. and 120-130 per cent. respectively. Although these are very good yields indeed, the economic feasibility of the process would depend upon the hydrogenated furanes being available at a low price, and it is doubtful whether this is yet the case. It may be noted in this connection that cheap tetrahydrofurane would also be of great interest as a solvent for synthetic resins (*see* THE CHEMICAL AGE, 1940, *43*, 1107, p. 120) and as an intermediate for butadiene rubber.

A New Adhesive

Urea-Formaldehyde Products Developed in U.S.

A NEW adhesive, now commercially available in the United States, will glue together two non-porous metals. Till now this process has been possible only under hot-presses and therefore out of reach of the layman. The new glue is a urea-formaldehyde product. It is sold in the form of a fine powder and ten parts of the product have to be mixed with six parts of water by weight. After two or three hours of application the bond is strong enough for light handling. After 48 hours it takes on waterproof characteristics and in two weeks the bond is complete. This is the first cold setting, one-part, resin adhesive to be produced in the United States.

The H. E. Armstrong Lecture

"Father of British Chemistry" Honoured

Dr. E. F. Armstrong

AN interesting occasion in the history of British chemistry is marked by the forthcoming meeting of the London Section of the Society of Chemical Industry, to be held next Monday in the rooms of the Chemical Society at Burlington House at 2 p.m. The occasion is the first of a series of lectures, to be delivered at irregular intervals by eminent chemists, designed to preserve the memory of Professor H. E. Armstrong, who has justly been described in recent years as the "Father of British Chemistry."

The choice of Professor Armstrong's famous son, Dr. E. F. Armstrong, as the first lecturer, was an obvious step. Dr. Armstrong is a past President of the Society of Chemical Industry and his lecture, which on this first occasion will be predominantly biographical, will be given with an authority and accuracy that would be denied to any other author. He is at present Chief Chemical Adviser to the Ministry of Home Security.

When Professor Armstrong died on July 13, 1937, at the age of 89, he was senior Fellow of the Royal Society, and probably the most prominent figure in British chemistry. His career, throughout his long life, was one of unremitting service to his chosen profession, notably in the promotion of the adequate teaching of science, and to the end of his days he was a great power among the majority of the scientific societies responsible for the dissemination of new knowledge in their several subjects. It is, however, even more for his own power and influence as a teacher that he should be remembered. A very high proportion of the more noted British chemists of the last 30 years acknowledge the inspiration derived as students or research workers from Armstrong's encouragement and guidance. Not the least part of his influence was derived from his own achievements in the world of chemical science, his vigorous, original, and independent personality, and his forceful method of expressing himself both in speech and in writing.

For many years he was a regular contributor to THE CHEMICAL AGE, and readers will remember his stimulating and entertaining articles, written under the rubric of "At the Sign of the Cheshire Cat." The following are two examples of the comments that he made in our columns within the last twelve months of his life.

Writing of the British Association's meeting in Blackpool in THE CHEMICAL AGE for October 3, 1936, he said:

"What went the Association out to for to do? It is clearly between the Devil and the Deep Sea; the Council can only talk in terms of worn-out Tommyrotics. Several months ago a programme was issued in which attention was called to the original object of the Association:

. . . *To obtain more general attention for the objects of science, and the removal of any disadvantages of a public kind which may impede its progress.* . . .

A century or more ago when this article was framed original workers lived all but isolated lives. . . . During the Victorian period the Association did a great work in promoting the interests of scientific students: it is doubtful if it has had the least useful effect upon scientific progress in the interval. The agencies are now almost too numerous. What happens at the Association meetings does not reach the public, except in tit-bit form; the proceedings are not snappy enough to appeal to the average press-man."

Again, writing on the subject of chemical education in the issue of November 28, 1936: "I would deny at once the assumption made by H. G. Wells that all men can and will ultimately be able to think alike: that, in fact, a world Utopia is possible. Mr. Wells will never 'see it through.' Chemistry might as well be reduced to a single element; a single type of structure. His doctrines are of no practical value. He is an ancient number, in fact."

Canadian Chemists Confer

Secondary Resources and Personnel Discussed

CANADA'S chemists are wholeheartedly behind the plan of the Royal Society of Canada to determine and develop Canada's secondary natural resources. This was emphasised at the end-of-the-year meetings of the councils of the Canadian Institute of Chemistry, the Canadian Society of Chemical Industry, and the Canadian Association. As luncheon guests of the Montreal section, S.C.I., the three councils heard Dean J. J. O'Neill, of the McGill School of Graduate Studies, outline the Royal Society's proposal. He explained that secondary resources in mining areas should be developed to assure continued activity when primary resources had been exhausted.

The councils suggested a meeting of representatives of provincial and federal governments and technical societies to study the matter further. The meetings also discussed the federal government's proposal about efficient utilisation of technical personnel in Canada's war effort, dwelling particularly on the drafting of chemists in non-essential industries into the field of war industries. Further study will be given to the question by the Engineering Institute of Canada, the Canadian Institute of Chemistry, and the Canadian Institute of Mining and Metallurgy.

Presiding over the gatherings, the first that the councils have held in Montreal, were Mr. A. F. G. Cadenhead, Canadian Institute of Chemistry; Mr. E. Bartram, Society of Chemical Industry; and Dr. P. E. Gagnon, of Laval University, Quebec, Canadian Chemical Association; and Mr. J. S. Rovey, chairman of the Montreal Section, Society of Chemical Industry.

LECITHIN FOR PETROL STORAGE

Lecithin, a phosphatide that has lately been produced economically from soya beans, is an effective deterrent to the deterioration of petrol on exposure to sunlight. This was reported at the annual meeting of the American Petroleum Institute in a paper by H. V. Rees, W. S. Quimby, and J. C. D. Oosterhout, of The Texas Company. Addition of lecithin also has an important corollary effect, they stated, in decreasing the corrosion of storage tanks and galvanised drums, particularly where the product stored is a highly-leaded aviation fuel. Corrosion in aluminium fuel tanks also is reduced.

Personal Notes

SIR CLIVE BAILLIEU, following his appointment as director-general of the British Purchasing Commission, has resigned his position as chairman of the Imperial Smelting Corporation, the Zinc Corporation, and New Broken Hill Consolidated, Limited. MR. JOHN GOVETT has been appointed in his place.

OBITUARY

MR. GEORGE HERBERT ROGERSON, for 45 years principal of Rogerson's Dye and Chemical Works, Lancaster, has died aged 78.

MR. CHARLES HENRY HAMILTON, formerly chairman of William Gossage and Co., soap manufacturers, Widnes, died last week, aged 67.

New Control Orders

Coal Tar Distillation

AN order controlling coal tar, which comes into force on February 1, has been signed by the Secretary for Mines.

Under the Order, the acquisition of crude coal tar by distillers will be controlled by licence immediately. The Secretary for Mines has power to order control by licence of the disposal by distillers of any of the primary products of the distillation of crude tar. Control has been found necessary because of the great importance of coal-tar products in the national war effort.

Benzol and toluene are not covered by the Order.

Preparation of Triazines

Melamine, Ammeline, and Ammelide from Dicyandiamide

MELAMINE (or 2,4,6-triaminotriazine) was first made on the commercial scale by pressure reaction of dicyandiamide with anhydrous ammonia. More recently it has been found possible to carry out the conversion at normal pressure and so avoid the use of stirring autoclaves. One method is based on the discovery that melamine is readily formed when dicyandiamide is heated with a strong alkali such as caustic potash in a high-boiling organic liquid. This is covered by U.S.P. 2,206,005 of the American Cyanamid Co., in which it is claimed that an 80 per cent. yield of melamine (based on the weight of starting material) is obtained when a dispersion of 50 parts dicyandiamide and 5 parts 85 per cent. caustic potash in 100 parts benzyl alcohol is agitated at 170°-200° C. The reaction also results in formation of some 20 per cent. of ammeline (or 2-hydroxy-4,6-diamino-1,3,5-triazine). A still lower reaction temperature is employed in the process of U.S.P. 2,206,603 (American Cyanamid Co.) where the dicyandiamide is heated in the absence of caustic alkali, but in the presence of zinc chloride. Here 440 parts dicyandiamide were slowly stirred into a solution of 25 parts zinc chloride in 500 parts ethylene glycol which had been brought to 100° C. After heating at 100°-110° C. for 2½ hrs., a crystal mass of melamine was formed. The yield was considerably less than in the first-mentioned process, only amounting to 35 per cent. of the weight of dicyandiamide. It is interesting to note, however, that the process can be modified to give high yields of other aminotriazines. On refluxing 100 parts dicyandiamide in 200 parts water in presence of 4 parts zinc chloride for 2 hrs., the insoluble product was a mixture of ammeline and ammelide. The latter is 2,4-dihydroxy-6-amino-1,3,5-triazine. Like melamine itself, the hydroxy-compounds condense with aldehydes to give thermosetting resins.

Alternative types of valves for handling most of the better known industrial fluids are described and illustrated in the attractively printed Handbook 740 issued by SAUNDERS VALVE COMPANY, LTD., Cwmbran, Newport, Monmouthshire.

The Political Method

Sir Ernest Benn Attacks the Bureaucrats

JUST as the mycologist watches the mycelium of a fungus spreading from root to root or from stem to stem of a plant on which it has gained hold, so does Sir Ernest Benn, in his latest pamphlet,* examine the parasitic growths of bureaucracy spreading from branch to branch of the national organism. His approach to the consideration of the problem of this parasitism is as scientific as that of the mycologist. He realises that it is useless to jeer or complain because bureaucrats behave as such; it is no good, in fact, making a fuss because a parasite is parasitic. The trouble is that just as wrong methods of cultivation are apt to bring the scourge of mould or blight upon a crop, so a faulty outlook on national affairs can lead to the dead-hand method of bureaucratic control—the "Political Method," in short.

Sir Ernest Benn, as is well known, is an advocate of the method of free competition in buying and selling; with all its attendant evils, he contends, it is better than the grandmotherly system of official supervision of every transaction. And there is a great deal in what he says. Putting it at the lowest level, it is better to get *something* done, even if a certain amount of harm to some parties may be caused, than to get *nothing* done at all, whereby all the parties concerned suffer harm. One of the main ostensible objectives of bureaucracy in business affairs is that the bureaucrats, being *hors concours*, as it were, should act as a safeguard against unfair trade; that they should, in fact, eliminate the profiteer. Even supposing they did this (a more than doubtful matter, as Sir Ernest demonstrates), their method is such that they eliminate everyone else, too. Sir Ernest cites the case of the trade sanctions against Italy. Instead of leaving the enforcement of the sanctions to the dockyard police, the "political method" people introduced a system of controls whereby, it is true, trade with Italy was reduced to a minimum, but as a result of which a numbing paralysis affected all other export trade as well.

During and after the last war, Sir Ernest Benn himself served as a temporary bureaucrat, and his experiences of those days enable him to write with inside knowledge of the system. Some of his more infuriating experiences he relates with his customary verve and incisiveness. But we have bigger and better bureaucrats since then, and wider and more complicated controls; and our author is able to point out some truly awful examples of their effect on our national life to-day.

There is a ray of hope, however. The dire necessity of prosecuting Hitler's war with vigour has caused the entangling parasite threads to be hewn through in one or two vital instances; and there is evidence that some, at any rate, of those who control our bureaucrats have not forgotten the forthright principles of Tom Paine and "The Rights of Man."

* *The Political Method*. By Sir Ernest J.P. Benn. London: The Individualist Bookshop. Sixpence.

INDUSTRIAL DRYING

The Annual Trade Review recently published by the *Manchester Guardian* contains, as is wise and customary, a valuable series of articles on the progress of commerce, especially as affecting Lancashire. An uncommon and interesting feature is a short review entitled "Industrial Drying," which gives a broad outline of the different forms of dryers applicable to various products. We read, however, that in some industries drying is still carried out by haphazard and inefficient methods. In view of the highly efficient apparatus for the drying of chemical and allied products that is obtainable, as THE CHEMICAL AGE has often pointed out, this is a little surprising; and any concerns that have not taken advantage of the modern scientific progress in the art and economics of drying would do well to consult the article and follow its advice.

General News

A SPECIFICATION, numbered D.T.D. 911, and superseding S.I.S. 85, has been issued by the Ministry of Aircraft Production for "The Protection of Magnesium Rich Alloy Parts against Corrosion," price 1s.

MONSANTO CHEMICALS, LTD., Ruabon, Denbighshire, have distributed copies of their useful wall calendar for 1941. In these days this calendar, showing as it does three months at a time, is very acceptable.

"PRODUCER GAS FOR ROAD VEHICLES" was the subject of an address given by Dr. A. A. Crawford, of Ashington Coal Company, to members of the Newcastle section of the Society of Chemical Industry following a luncheon at the Royal Turk's Head Hotel, Newcastle, on January 23.

A NEW EDITION of the "British Pharmacopœia" will shortly be produced. This publication is brought up to date every ten years, and an interesting coincidence is that the latest edition will appear during a year in which both the Pharmaceutical Society and the Chemical Society celebrate their centenary.

TO CREATE A MARKET for heavy brass disposal, the Non-Ferrous Metals Control is prepared to buy brass ingots of a defined analysis at £35 per ton f.o.r. makers' works. The copper content is allowed to vary from 62 to 65 per cent., and reasonable latitude is given for tin, lead, iron and other elements.

A SCIENCE TEACHER in a Midlands technical school, unwilling to pay the high price now ruling for mercury, is reported to have set the laboratory boy to see what he could recover from the trap under the laboratory sink. He spent a profitable two hours, at the end of which the laboratory and the country were the richer by over nine pounds of quicksilver, worth over £16. Other technical schools, please note.

AVAILABLE SUPPLIES OF POTASH fertiliser in this country are being reserved mainly for crops which must have potash, such as potatoes and flax. Agricultural salt is being recommended in place of potash for sugar-beet and mangolds, but Mr. G. A. Cowie, chief agricultural adviser of Potash, Ltd., has issued a strong warning against the use of salt for potatoes, as experiments have shown that it may adversely affect both quantity and quality of this crop.

THE BRITISH STANDARDS INSTITUTION has issued a British Standard Method for the Biological Assay of Vitamin D_3 by the Chick Method (B.S.911). The antirachitic vitamin D_3 activity of an oil or vitamin D concentrate used for feeding livestock is estimated by comparing it by a recognised method with the antirachitic activity of the "Provisional Standard Preparation" of vitamin D_3, when so estimated is expressed in B.S.I. units per gramme.

OUR CONTEMPORARY, *The Gas World*, last week carried an appeal to readers, by Mr. Alexander Duckham, of Alexander Duckham & Co., Ltd., on behalf of the R.A.F. Benevolent Fund, and we feel that we also are justified in bringing this very worthy cause to the notice of our readers. An investment of £1,000,000 is aimed at, to bring the income up to at least £50,000. Lord Nuffield, with his usual sympathy and generosity, has contributed £250,000, and in addition £60,000 has already been collected, leaving £700,000 still required. The appeal is addressed primarily to the Gas Industry, but any other contributions will be gratefully acknowledged. They should be made payable to the R.A.F. Benevolent Fund and sent to Mr. Duckham at 16 Cannon Street, E.C.4.

Foreign News

PENDING THE AVAILABILITY of adequate supplies of fruit and vegetables, all German coalminers, it is reported, will be supplied daily from February 1 with two Vitamin C tabloids.

IMPORTS OF PAINT PRODUCTS into the Union of South Africa during the first half of 1940 were 32 per cent. higher in value than for the same period of 1939.

THE HIGHER PRICE of Pennsylvania crude oil, representing Bradford and Alleghany, was raised on January 27 from $2.15 to $2.30 per barrel. The lower price—for Corning crude—remains unchanged at $1.12 per barrel.

A NOTIFICATION IN THE *Federated Malay States Gazette* of November 1 prohibits the importation into the F.M.S. of hydrochloric acid, and of any coagulants containing hydrochloric acid, except under permit.

From Week to Week

THE CHAIRMAN of the Mount Morgan Mines, Brisbane, stated recently that an enormous increase in the output of Australian copper is expected to result from the opening of new plant. He added that the new plant would enable the company to increase production by 50 per cent. over last year.

THE AMERICAN BUREAU OF METAL STATISTICS report stocks of refined lead at the end of December as 40,900 tons, compared with 35,800 at the end of November. Production during the month was 61,800, against 57,900, while dispatches were 56,800, against 62,500.

THE GOVERNMENT OF MYSORE has approved the establishment of a factory to manufacture sodium bichromate to supplement the current shortage in the Indian dyeing and tanning industries due to the war. The question of manufacturing bichromates from chrome ore available in Mysore State has been under consideration for some time. Machinery and equipment for the plant will be obtained locally.

EXPERIMENTS MADE on young thinnings from teak plantations in Trinidad, with a view to ascertaining whether they could be utilised in the manufacture of pulp and paper, are reported locally. Tests carried out at the Imperial Institute in London led to the conclusion that, owing to the poor fibre length of the samples, the soda process was the only one suitable for commercial treatment. Such woods find little outlet in paper making except in the form of bleached soda pulps, and young teak, though readily digested by the soda process, does not yield an easily bleachable pulp.

Forthcoming Events

THE FIRST H. E. ARMSTRONG LECTURE of the Society of Chemical Industry will be delivered by Dr. E. F. Armstrong, on February 3 at 2 p.m., in the rooms of the Chemical Society, Burlington House, Piccadilly, W.1 (*see* p. 72.)

AT THE MEETING of the Road and Building Materials Group of the Society of Chemical Industry on February 4, at 2 p.m., Dr. F. H. Garner will read a paper on "Studies of Bituminous Mixture in Road Testing Machines," written by Mr. C. Mack, of the Technical and Research Department of Imperial Oil, Ltd., of Sarnia, Ontario.

A MEETING OF THE Society of Public Analysts and Other Analytical Chemists will be held in the Chemical Society's rooms, Burlington House, Piccadilly, London, on February 5, at 2.30 p.m., when the following papers will be presented and discussed: "The Melting Range of Fats," by K. A. Williams B.Sc., F.I.C.; "The Interference of Selenium and Tellurium in the Determination of Arsenic by the Hypophosphorous Method," by H. J. G. Challis, A.I.C.; "The Determination of Zinc in Magnesium and Nickel Alloys," by E. Q. Laws, B.Sc., F.I.C.; and "Studies in Internal Electrolysis"; and "The Determination of Small Quantities of Cobalt in presence of Zinc," by J. G. Fife, M.Sc., F.I.C.

THE NEXT MEETING of the Nottingham Section of the Society of Chemical Industry will be held at the Welbeck Hotel, Nottingham, on February 6, at 6.45 p.m., when Dr. W. F. Short will read a paper on "Chemotherapy in Relation to Dyestuffs and Their Intermediates."

THE NEXT MEETING of the Fuel Luncheon Club will take place at the Connaught Rooms, Great Queen Street, Kingsway, W.C.2, on February 6, at 12.40 for 1.10 p.m., when the after-luncheon address on "The War Damage Bill and Its Relation to the Fuel Industries" will be given by Capt. the Rt. Honorable H. F. C. Crookshank, P.C., M.P., Parliamentary Secretary to the Treasury.

A JOINT MEETING of the Food Group and the Manchester Section of the Society of Chemical Industry will be held on February 7 at 1.30 p.m. in the Reynolds Hall, College of Technology, Manchester, when Mr. W. A. Richardson will present a paper on "The Swelling Behaviour of Starches."

ON FEBRUARY 8, at 3 p.m., a meeting of the South Wales Section of the Society of Chemical Industry will be held in the Chemistry Theatre, University College, Swansea. Dr. J. A. Sugden, of the Steatite and Porcelain Products Co., Ltd., will lecture on "The Development of Ceramic Electrical Insulating Materials," and a discussion following the paper will be opened by Mr. G. P. Britton.

Metallurgical Section

Published the first Saturday in the month.

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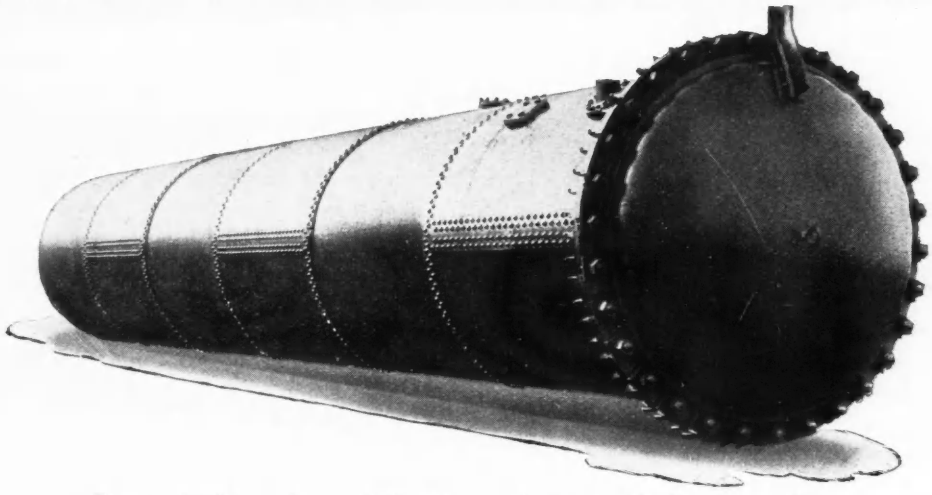

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Hot-Tinning

A Useful Technical Publication

UNDER the above title, the Tin Research Institute has issued its "Publication No. 102," a comprehensive account of the different processes used in hot-tinning, by C. E. Homer, B.Sc., Ph.D. Explanations of the various factors influencing the quality of the coatings produced are likewise included. In the introductory paragraphs the applicability and the limitations of the process are discussed; hot-dipping is suitable for mild and carbon steels, copper and many copper alloys, and for cast iron and certain alloy steels when properly prepared. An attractive feature of the process is that only a few seconds' immersion in molten tin is required to produce a brilliant coating that requires nothing more than washing or wiping to finish it. The preliminary processes are fully described. For degreasing, the three principal agents used are organic solvents, alkaline solutions, and heat. For pickling, the two acids most used are cold hydrochloric and hot sulphuric, the latter very often being used with an inhibitor; nitric acid is effective with some difficult steels.

Methods of Tinning

Various types of tinning baths are discussed, with their operating temperatures and their maintenance in continuous operation. The simplest process consists of a single bath of molten tin, using zinc chloride as a flux; for a more attractive-looking coating, a dip in a second bath covered with a deep layer of oil is employed. A method suitable for thin articles is salammoniac tinning at comparatively high temperatures. Grease-tinning is an old established process in which the articles are immersed in grease before entering the first tinning bath; this method avoids any risk of subsequent discoloration or corrosion from spots of flux remaining on the finished articles, and there is less risk of rusting during storage; from the operating point of view, this process is especially attractive because contamination by iron of the molten tin in the bath occurs only very slowly.

The brightness of coatings is dependent chiefly upon the rapidity with which they solidify; when cooling is too slow the tin may oxidise to a yellow colour and the coating may drain so much that the upper parts of the article are insufficiently coated; on the other hand, cooling too rapidly is liable to cause a frosty-looking coat. The thickness of the metal article must be taken into account; light articles may be best when cooled in air, while heavier-gauge articles may need quenching. Another factor is the purity of the tin; certain impurities in the tin may cause a spangle like that on galvanised articles; the notion that the brightest coatings contain traces of some other metal is not correct.

The tinning of copper by hot-dipping follows the procedure outlined for steel, but there are some important differences in detail which are described in the second part of the book. Many practical difficulties and points of control are dealt with, *e.g.*, the contamination of the tinning bath with dissolved iron or copper; and purifying methods are described.

Ten diagrams and illustrations and a useful list of references complete a booklet that should be helpful to practical tinners and to works control staff.

Metallurgical Section

February 1, 1941

RECLAIMING ANTIMONY FROM WASTE

An Economic Use for Speiss

by A. G. AREND

THE value of antimony differs from that of many other metals in that it assumes greater importance in wartime, especially as this country is very largely, if not almost entirely, dependent upon imported supplies. Under peace conditions, the industrial consumption of antimony is chiefly accounted for by the production of white bearing alloys, type metals, and other special alloys of low melting point, while its salts are used in the ceramic, paint, rubber, dyeing, match and firework industries. In present circumstances, however, the metal is an important factor in the production of projectiles, because of the way in which the brittle particles can scatter.

While a fair amount of antimonial lead material is locally available, the pure ore has to be imported from a long distance, in fact from China. To understand this better, a scrutiny of export statistics will give some indication of the position, although owing to lack of data no absolute statistics of the world's production are available (*Metal Statistics*). In 1929 China provided 22,401 metric tons out of a world-total of 31,586 tons, *i.e.*, China produced two-thirds of the world's output; but in 1935 Chinese output had fallen to 17,700 tons, while the total for the world was approximately 25,700 tons. During this interval, although America possessed little domestic ore, a large industry of recovering antimony from secondary material was instituted there, accounting in 1935 for some 9,600 tons.

Antimony is recovered from secondary products in this country on a modified scale, but it has to be admitted that numerous odd waste products from different metallurgical operations exist, while little or no attempts are made to reclaim the metal. Smelting works which are already engaged on treating ores and residues have the plant available to deal with materials such as flue-dust, liquation drosses, and refinery slags, but speiss is a noticeable exception. A small use can be found for speiss as a means of providing arsenic for certain odd processes, but on the whole, the majority of it is not generally dealt with, and is left to accumulate as a waste. Speiss is obtained in the smelting of nickel, copper, and tin ores and residues, and varies very widely in composition as a result, and this has been largely responsible for its neglect.

Efforts are made by different smelters to try and avoid the formation of more speiss than absolutely essential, so that this loss will be reduced to a minimum. The antimony content may be anything from 10 to 15 per cent., and it would be a profitable proposition to recover the metal, but for the complexities of the impurities and other constituents that are present.

Typical Speiss Contents

A typical nickel speiss has the composition: 35.2 per cent. nickel, 17.2 per cent. copper, 18.7 per cent. arsenic, 11.1 per cent. antimony, 13.4 per cent. iron, 1.6 per cent. lead, 2.3 per cent. sulphur, but each of the constituents can vary by several per cent. Thus the iron content may fall to under 8 per cent., and the lead content may rise to over 7 per cent. The same remarks apply to copper speiss, which forms in a different manner in both blast smelting and reverberatory smelting of copper ores and residues. The following is a typical copper speiss: 24.0 per cent. copper, 16.5 per cent. lead, 12.2 per cent. arsenic, 13.5 per cent. antimony, 25.2 per cent. iron, 3.2 per cent. nickel, 3.0 per cent. zinc, and 2.3 per cent. sulphur.

The type of speiss which accumulates in tin refineries varies between the two kinds of composition detailed, and there is always a certain varying proportion of cobalt, which, however, never appears in practice in the high percentages generally quoted in earlier metallurgical literature. In the smelting of tin residues, the desire to prevent any undue presence of sulphur causes a somewhat larger proportion of white-metal or speiss in the incipient stages. Were this to be sulphurised it would be possible to reduce the copper, and always keep it down to a low level.

After an investigation of a very exhaustive range of speiss compositions, it was revealed that the antimony content rarely fell below 8 per cent., and more often averaged about 12 per cent. When it is recalled that other by-products, such as flue dusts, drosses, and liquation residues, contain little more, and seldom exceed 20 per cent. of antimony, it will be observed that a small, but otherwise useful source of antimony exists in this country, which, because of working complications, is at present ignored. Should the present conditions make any shortage of antimony apparent, all speisses collected from different by-products could be treated, and thus ensure a certain measure of independence. The copper and nickel contents can be recovered during the working of the same process, but it must be mentioned that there are numerous poorer speisses rich in iron which are correspondingly lower in these metals, and which still maintain the same high antimony content.

The expression "by-products" must not be confused with "waste products," because by-products such as liquation residues, flue dusts, refinery slags, etc., are practically always worked, whereas speiss is ignored, and is properly represented as a waste.

Fusion and Wet Extraction

The speiss is fused in a small electric furnace together with pyrites, which, by adding appreciably to the sulphur content in the absence of air, gives a concentration of matte, and enriched speiss, and evolves the surplus arsenic in the form of the volatile arsenic sulphide. With most speisses it is necessary to make the sulphur addition so that the mixture will ultimately contain some 38 per cent. of sulphur, but this has to be varied according to the changing composition. When the correct percentage of pyrites has been found by experiment, the matte should not contain more than 0.03 per cent. antimony, as the latter has been concentrated in the fresh speiss to the extent of some 20 per cent. The copper speiss in the example quoted above was mixed with 1½ times its weight of pyrites, and gave this result. No other addition agents or fluxes are required, and in some cases upwards of 90 per cent. of copper can be removed in this manner.

The furnace used was copied from one originally described in *Metall und Erz* in 1919, but which was improved on by substituting a silica lining and more suitable bottom connection. The hearth is of oval shape, with water-cooled leads, connected to a bottom plate of copper covered with several

inches of carbon material tamped into the bottom with pitch and burned hard. Graphite electrodes, one for each hearth, enter the small roof through asbestos glands, and are operated by double square-threaded screws. At one side of this hearth, a refractory pipe-line leads off the volatile products, chiefly arsenic sulphide, to a condensing chamber, where they are collected.

The condensing chamber consists simply of a square construction equipped with a considerable number of baffles, upon which the arsenic sulphide alights, and which allows of the easy recovery of the latter when the smelting has been completed. The products of the smelting are a rich matte, slag, and a concentrated speiss chiefly composed of nickel antimonide, and these are easily separated by the method of tapping. After the speiss has been crushed to pass through a 16-mesh sieve, it is mixed with powdered sodium sulphate and coal, and fused at a dull red heat to convert the antimony to sodium sulphantimonite. The method described in *Metall und Erz* was found to be at fault, as it was proposed to have a molten bath of sodium sulphate and coal awaiting the tapping-in of molten speiss, which can never react with the same precision as where the finely divided materials are charged together. Molten speiss possesses much too high a specific gravity to give more than a very imperfect reaction, and one which is slow to perform. The furnace used for this treatment is a small reverberatory hearth, and the mass is simply mixed up until all has become fluid, when it is tapped into iron moulds. The product is then broken to small pieces by passing through a jaw-breaker, after which it is finely pulverised in a small ball-mill, and ejected into an iron tank of boiling water. This dissolves out the easily soluble antimony salts, to the extent of 99.5 per cent., while the whole of the nickel, copper, and other metals remain as insoluble sulphides. In this respect the process is unique, as it is but rarely that so complete a separation can be effected; the reason being that the only soluble base metals are represented by tin, arsenic, and antimony. Tin need not be present to any appreciable extent, as it is generally drawn off by contact with lead, and further forms a volatile sulphide like that of arsenic in the electric furnace, whereas the antimony remains stable.

Depositing Antimony by Electrolysis

As the residue is slimy and difficult to filter with precision, the liquor is left to stand for some little time to allow the insoluble sulphides to separate out, after which it is drawn through a vacuum filter. The precipitate was originally roasted and smelted with sand to give a crude copper-nickel alloy which was disposed of in this condition, while lead was slagged off, but this method was improved on by separating the constituents. The antimonial liquors are run into electrolysis vats—rectangular iron tanks equipped with sheet iron cathodes and lead anodes. As the density diminishes during the treatment, pipe-lines are used to run the liquor in at the bottom of the tanks, following the original process. The iron sides of the tanks can be made to act as additional cathodes, to facilitate the deposition of antimony. The current applied has to be reduced as antimony is removed from the electrolyte, since otherwise it would be dissipated wastefully, and in the same manner the process is continued only until the antimony content has fallen to 1 per cent., below which deposition is uneconomical. As it is, the somewhat promiscuous and continual necessity for altering the current results in an efficiency seldom exceeding 50 per cent. in actual practice, although much higher figures have been cited elsewhere. Recently, by means of applying control instruments which automatically alter the current input according to the density of the electrolyte as it becomes reduced, it has been claimed that high current efficiency has been attained. An alternative system consists of connecting up additional baths, or connecting a few baths in parallel at the end of the series, but the control-instrument system has outclassed such methods. Current density is not increased beyond 115 amps. per sq. metre to start with, and has to be reduced to under 50 amps. at the final stages. Although the process has been criticised because of the powdery nature of part of the product, instead of a hard genuinely metallic deposit obtaining, it has been persevered with, particularly because a certain proportion of iron is invariably present, and there is in any case no alternative but to melt the metal subsequently. When the electrolysis first commences, the antimony comes down in lustrous scales, but as the amperage comes to be decreased, the more powdery material is deposited, some of it eventually falling to the bottom of the tank, instead of being retained on the cathode. In removing the deposited antimony from the iron cathode, some small modicum of iron is again incorporated, but except for this, the product is the pure metal. After removing the deposit from the cathodes, the black mass is gently dried in an oven, mixed with a very small proportion of antimony sulphide, melted, and poured into plate-moulds. This last addition is calculated on the percentage of iron present, so that little excess of sulphur need be present to form iron sulphide. As the specific gravity of antimony is 6.6, and that of iron sulphide 4.8, the impurity is given good opportunity to separate as a top layer, without impoverishing the antimony. Whereas antimony obtained by pyrometallurgical methods contains small quantities of lead, copper, arsenic, and sulphur, besides iron, the electrolytic product obtained by the foregoing process contains upwards of 99.9 per cent. antimony, and the only impurity is little more than a trace of iron.

Nazi Metallurgical Plans

Economic Grip on Norway and France

THE metallurgical industries of the Nazi-occupied countries are being subjected to a continuous process of exploitation on the part of the invader. Recent reports about conditions in Norway and France indicate how the pressure is being applied.

The Swedish newspaper *Nya Dagligt Allehanda* reports that a new company has been formed in Berlin to exploit the aluminium resources of Norway with the title of "Nordiska Aluminium" and RM 20,000,000 capital. The founders are "mainly people interested in the aircraft industry."

At the same time a "close linking up of the metal-working industries of Germany and France" is reported in Vichy as being now under discussion. French industrial circles are said to expect a strong revival of activity from this "co-operation" in the German war effort on the grounds that "Germany is still suffering from a lack of workmen, while French industry has so far been unable to cope with unemployment." Germany is said to be willing to barter raw materials for French labour. It is clear that, with Germany in control of raw materials and labour supplies, the French industry would be in a completely subordinate position.

Another method by which German firms are able to control the production of firms in occupied countries which were formerly their competitors is exemplified by the merging of the Belgian Steel Company, Société Commerciale d'Ougrée, S.A., with the German firm of Otto Wolff. The Belgian company operated not only the blast furnaces at Ougrée in the Liége industrial district, but also those at Rodange in Luxembourg, and commanded its own sources of iron ore in France and Morocco. The "merging" of the two firms relieves the Germans, ostensibly, of the stigma of expropriation.

U.S. TIN PURCHASE

The purchase by the United States of 40,000 tons of tin from the Far East has been announced in Washington by Mr. Jesse Jones, the U.S. Secretary of Commerce, states a B.U.P. message. Fifteen thousand tons had been delivered; another 15,000 tons was awaiting shipment; and the remainder was on its way. The United States was also planning to buy 90,000 tons of tin from Bolivia, Mr. Jones added, at a total cost of £25,000,000.

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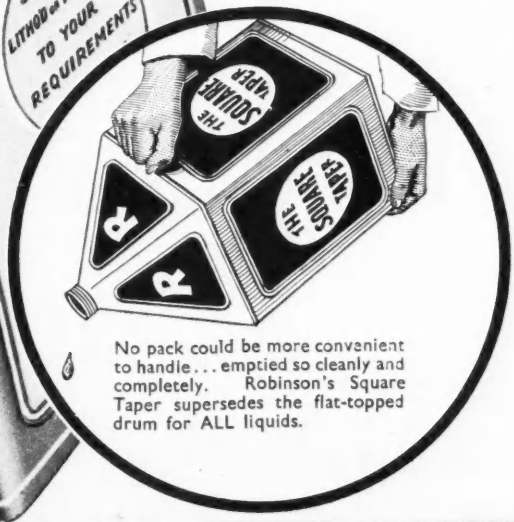

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Commercial Intelligence

The following are taken from printed reports, but we cannot be responsible for errors that may occur.

County Court Judgments

ROBINSON, EDWD., 35 Merlin Road, Middlesbrough, chemical worker. (C.C.J., 25/1/41). £15 9s. 0d. November 29.

HANLYN & HOUBEN, LTD., R.O., 110 Fleet Street, E.C.4, manufacturing chemist. (C.C.J., 25/1/41.) £19 0s. 2d. November 25.

Receivership

CELLULOSE COMPOSITIONS, Magneto Works, Trumpington, Cambridge. (R., 1/2/41.) Wm. L. Dominy, of Sussex House, Hobson Street, Cambridge, was appointed receiver on January 15, under powers contained in debenture dated March 11, 1938.

Company News

R. B. Pullin & Co., scientific instrument manufacturers, announce a final dividend of 15 per cent., making a total of 20 per cent. for the year.

Roche Products, Ltd., pharmaceutical preparations manufacturers, Broadwater Road, Welwyn Garden City, Herts, have increased their nominal capital beyond the registered capital of £20,000 by the addition of £50,000 in 50,000 ordinary shares of £1 each.

Dewalco, Ltd., manufacturers of compositions for sealing of glass and metal containers, etc., 3 St. James Square, W.C.1, announce that the name of the company was changed to **Dalex, Ltd.,** on January 13.

The Deason Chemical Company (1938), Ltd., 5a Princes' Way, Team Valley Trading Estate, Gateshead-on-Tyne, announce that the title of their company is changed, as from January 4, to **Barluze (Products), Ltd.**

Messrs. N. M. Rothschild and Sons announce that coupon No. 30, due February 1, 1941, of the 4½ per cent. debentures and coupon No. 4, due February 1, 1941, of the 3½ per cent. debentures of **N.V. de Bataafsche Petroleum Maatschappij** may now be presented to them for payment in sterling at the rate of guilders 7.60 to £1. Coupons must be handed in personally or by a London banker or other agent.

Chemical Trade Inquiries

Chile.—An agent established at Santiago wishes to obtain the representation of United Kingdom manufacturers of chemical products. (Ref. No. 22.)

Iraq.—An agent established at Baghdad wishes to obtain the representation of United Kingdom manufacturers of chemicals and dyes. (Ref. No. 17.)

Portugal.—A firm of agents established at Lisbon wishes to obtain the representation of United Kingdom manufacturers of chemical products. (Ref. No. 16.)

Chemical and Allied Stocks and Shares

SUBDUED and moderately reactionary conditions have ruled in most sections of the Stock Exchange, owing to a decline in the volume of business and a tendency for profit-taking to develop in industrial securities following their recent improvement in value. Markets have been influenced by a disposition to await news as to the Government's plans for the mobilisation of man-power for war work, while it is being pointed out that the Budget is likely to bring a further increase in taxation. There are, however, hopes that E.P.T. may be modified in various respects, because in some instances it bears so heavily on earnings that a point is being reached when it will be difficult to make adequate allocations for depreciation and other essential provisions. Many financial results issued this year will, of course, be the first to reflect E.P.T. at 100 per cent. for a full twelve months.

Many important reports and dividend announcements fall to be issued in March, including the I.C.I. figures for 1940. As compared with a week ago, Imperial Chemical ordinary were little changed at 30s. 1½d., but the preference units were easier at 33s. It may be recalled that in respect of 1940 the interim dividend has been maintained at 3 per cent. and the market is continuing to look for a final dividend of 5 per cent., thus maintaining the total at 8 per cent.; in 1939 a dividend of 12½ per cent. was earned, but as usual, a good proportion of the profits was placed to reserve funds. Courtaulds have shown a fairly steady appearance at 32s. 9d. The final dividend announcement of this important company can also be expected in March, and estimates of the total continue to range from 8 per cent. to 10 per cent., which would compare with the 7 per cent. paid for 1939. The interim payment for 1940 was raised from 2 per cent. to 2½ per cent. Associated Cement have been firm at 60s. This company's 1940 figures are also due in March, and market estimates of the dividend are for 12½ per cent. or 15 per cent. Borax Consolidated deferred units at 28s. 6d. have held their recent rise on hopes that the forthcoming results may show the maintenance of the distribution at 7½ per cent.

Following their recent advance, Turner & Newall reacted moderately to 69s. 4½d., but Dunlop Rubber remained in request, and at the time of writing are around 34s. British Aluminium were steady at 42s. 6d. and British Oxygen were 66s. 10½d. On the other hand, Lever & Unilever became easier at 24s. 1½d., although the various classes of preference units were well maintained in price. General Refractories remained firmer at 8s. 4½d. on hopes of better results. Pinchin Johnson at 20s. 3d. lost part of their recent rally, but International Paint held their rise to 77s. 6d. and Lewis Berger again changed hands up to 42s. 6d. In accordance with the easier tendency on the Stock Exchange, Distillers ordinary units went back to 66s. 3d. but on the other hand, Nairn & Greenwich were firm at 51s. 3d. and Barry & Staines were 27s. Where changed, iron and steel securities lost a few pence; Stewarts & Lloyds were 43s., and Tube Investments 91s.

In other directions, B. Laporte changed hands up to 56s. 3d. Greeff-Chemicals Holdings 5s. units were around 6s., while Monsanto Chemicals 5½ per cent. preference shares continued to be quoted at 22s. 6d. British Drug were around 22s. 6d. and William Blythe 3s. ordinary shares have transferred at 5s. 3d. Imperial Smelting held their improvement to 11s. 3d. Wall Paper deferred units were easier at 22s. 10½d. and rather lower prices ruled for Murex. Cerebos and Reckitt & Sons ordinary units remained firmly held, and are unchanged in price at the time of writing. Burmah Oil, "Shell" and Anglo-Iranian lost part of last week's improvement; other oil shares were also slightly lower this week in accordance with the prevailing trend on the Stock Exchange.

British Chemical Prices

Market Reports

STEADY trading conditions are reported this week from all sections of the general chemical market and values are very firm, although there have been few, if any, actual changes in quotations from those recorded last week. A fairly satisfactory volume of contract renewal business has been replaced during the past few weeks and in the aggregate is not much below the corresponding period of a year ago. The demand for chemicals both for home and export continues active, and makers' deliveries are well up to expectations. In the market for coal tar products the chief feature is the strengthening of prices and this is particularly noticeable for such items as creosote, solvent naphtha and xylol. The market on the whole is fairly active.

MANCHESTER.—Firmness of prices remains a pretty general feature of the Manchester chemical market. Bleaching, dyeing and finishing chemicals have met with a steady demand this week from the textile industries, whilst most of the other consuming trades are giving a good account of themselves so far as deliveries against contracts are concerned. With regard to overseas business, inquiry has been on a moderate scale. Most of the tar products are on a distinctly firm basis and the recent rises in this section has been fully maintained. The light distillates especially, as well as creosote oil, are being taken up in good quantities.

GLASGOW.—There has been a slight improvement in business for general chemicals for home trade during the week, and also rather more inquiry for export. Prices generally continue very firm, though without many actual changes, but in several instances it is now stipulated that forward deliveries are to be charged at the price in operation at the time of delivery.

Price Changes

Ammonium Chloride.—MANCHESTER: Grey galvanising, £22 per ton.

Creosote.—Home trade, 5d. to 5½d. per gal., f.o.r., makers' works; exports, 6d. to 6½d. per gal., according to grade.

Dinitrobenzene.—8½d. per lb.

Glycerine.—Chemically pure, double distilled, 1.260 s.g., in tins, £3 15s. to £4 15s. per cwt., according to quantity; in drums, £3 7s. 6d. to £4 1s. Refined pale straw industrial, 5s. per cwt. less than chemically pure.

Naphtha.—Solvent, 90/160, 2s. 3d. to 2s. 6d. per gal.; heavy, 90/190, 1s. 7d. to 1s. 8d., naked at works. MANCHESTER: 90/160, 2s. 3d. to 2s. 6d.

Sodium Sulphate (Salt Cake).—MANCHESTER: About £4 2s. 6d. ex works.

Turpentine.—Spot, American, including tax, ex wharf, in barrels and ex discount: LONDON, nominal; GLASGOW, 70s. per cwt.; LIVERPOOL, 69s. per cwt.